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Assessing the impact of FAME and diesel fuel composition on stability and vehicle filter blocking

Gopalan, K.¹, Chuck, C.J.¹, Roy-Smith, C.², and Bannister, C.D.¹

¹ University of Bath, ² BP Formulated Products Technology

Abstract

In recent years, there has been an impetus in the automotive industry to develop newer diesel injection systems with a view to reducing fuel consumption and emissions. This development has led to hardware capable of higher pressures, typically up to 2500 bar. An increase in pressure will result in a corresponding increase in fuel temperature after compression with studies showing changes in fuel temperatures of up to 150 °C in 1000-2500 bar injection systems.

Until recently, the addition of Fatty Acid Methyl Esters, FAME, to diesel had been blamed for a number of fuel system durability issues such as injector deposits and fuel filter blocking. Despite a growing acceptance within the automotive and petrochemical industries that FAME is not solely to blame for diesel instability, there is a lack of published literature in the area, with many studies still focusing on FAME oxidation to explain deposit formation and hardware durability.

The majority of studies into diesel degradation are conducted under non-representative laboratory conditions, or are extrapolated from the deposits found in filters from vehicles with failed injectors. In this study, the cause of this degradation was investigated by using a novel High Pressure Common Rail (HPCR) non-firing rig designed to mimic a diesel common rail system, simulating realistic, albeit accelerated, operating conditions. The degree of deposition on the system fuel filter was monitored, for both petroleum diesel (B0), RF79 (B0), Bx (where x is percentage volume/volume of FAME) and surrogate diesel fuel components.

A systematic study of synthetic surrogates demonstrated that, as well as FAME, any base fuel component, under sufficiently high pressures and temperatures experienced in the HPCR are prone to degradation irrespective of the concentration of the component in the original fuel. The most unstable component acts as the instigator, thus promoting fuel oxidation. The other components in the fuel such as FAME, aromatic and cycloalkane portions will also oxidise and eventually polymerise to form solids blocking the filter. This also demonstrates that while a large body of work on the oxidative instability of biodiesel in the chemical laboratory is indicative of instability this does not mimic what is seen under more realistic vehicle conditions and the focus on FAME instability is misleading.

Introduction

In order to comply with Euro 6 emissions legislation, OEM's have adopted many new technologies, with combustion and fuel injections systems undergoing further development.[8] Diesel fuel injection pressures have seen an upward trend increasing significantly from

<700 bar to 2500 bar depending on the age and application of the engine. Light duty systems capable of achieving up to 3000 bar have been announced and are under development.[1, 2] An increase in pressure will result in a corresponding increase in fuel temperature after compression with studies showing changes in fuel temperatures of up to 150 °C in 1000-2500 bar injection systems.[3-7] Until recently, the addition of Fatty Acid Methyl Esters, FAME, to diesel had been blamed for a number of fuel system durability issues such as injector deposits and fuel filter blocking.[9-11] more recent studies have suggested that FAME is not the sole cause. Instead, the base diesel fuel can also undergo degradation at elevated pressures resulting in solid formation, even in the absence of FAME.[12] Other studies on B0 fuel stored for 1 year in barrels and aged under benchtop conditions (90°C and aeration) have shown that some diesel components (e.g. aromatics) present in base diesel (no FAME), also generate radical species resulting in diesel degradation.[13, 14]

Modern common rail fuel filters typically have a filtration rating of 2-5microns to enable the capture of macro-particles and a proportion of fine, insoluble, fuel deposits formed within the fuel circuit.[15] The deterioration of fuel and the formation of deposits are usually attributed to diesel fuel oxidation, with organic carbon being the predominant element though inorganic components have been found in the deposits also.[16] Many factors appear to affect diesel deposits. For example, a large body of work has been published to show that FAME has a tendency to oxidise and thus has traditionally been considered to be a major cause of fuel instability. Prolonged FAME degradation has been shown to lead to insoluble oligomer formation via a radical mechanism.[17-20]

Several methodologies have been developed to monitor the oxidation stability of commercial diesel and biodiesel fuels subjected to accelerated degradation under defined conditions. These tests include the measurement of the induction period (IP) using the revised Rancimat method (EN15751) and PetroOXY test (EN16091), iodine value, acid number, kinematic viscosity, peroxides, and insolubles.[10, 20] In a recent study, measurement of the IP by a PetroOxy test was used to follow the oxidation kinetics and the formed oxidation products. They found that this technique required only a small amount of sample and a short analysis time allowing for fast and highly reproducible results. It also provided flexibility in the operating conditions, and offered the possibility to study fuel kinetic oxidation and perform successive oxidation runs on the same sample.[21]

Although previous FAME investigations were conducted under non-representative laboratory conditions,[22] more recent work has demonstrated the use of actual fuel injection equipment (FIE) to stress the fuel in a more realistic in-vehicle environment. The degradation process in the FIE is accelerated by returning the fuel

passing through the injectors to the tank rather than being consumed through combustion.[3] It has been found that heated and injected return fuel to fuel tank has resulted in rapid degradation.[4, 23] Fuel recirculation occurs in a vehicle under real-world conditions due to fuel return circuits (from the high pressure pump, rail and injector spill). However, the validity of recirculating injected fuel within fuel systems rigs, and whether this introduces additional degradation issues in testing not normally experienced in-vehicle, remains ambiguous.

An investigation carried out on deposits found in injectors and fuel filters, from a range of vehicles that operated with higher injection pressures, demonstrated that a black (carbonaceous) polymeric organic solid deposited on the filter over time. These deposits were found to block the filter pores, reducing the number of pores available and thus reducing the performance of the fuel filters.[24] The deposits indicated possible precursor molecules, such as FAME and aromatic species from the parent diesel, which supported a complex fuel degradation mechanism involving more than just the biofuel content.

Despite a growing acceptance within the automotive and petrochemical industries that FAME is not solely to blame for diesel instability, there is a lack of published literature in the area, with many studies still focusing on FAME oxidation to explain deposit formation and hardware durability. In this study, a bespoke high-pressure common rail FIE rig was developed to test the effect of fuel composition on the degradation process, and investigate the factors influencing deposit formation on the fuel filter. The aim of this study was to replicate in-vehicle engine conditions and use model diesel surrogates, as well as B0 and B10 diesel fuel, to determine the impact on the rate of deposit formation of the relevant components of diesel fuel.

Experimental Setup and Procedures

The bespoke FIE test rig was designed to deliver accelerated diesel fuel degradation testing keeping the environment that the fuel experiences as close as possible to represent real-world conditions. The reaction parameters included variable injection pressure, an ability to heat the source fuel, control injection event and the ability to heat the injectors, through placement in a heating block.[12]

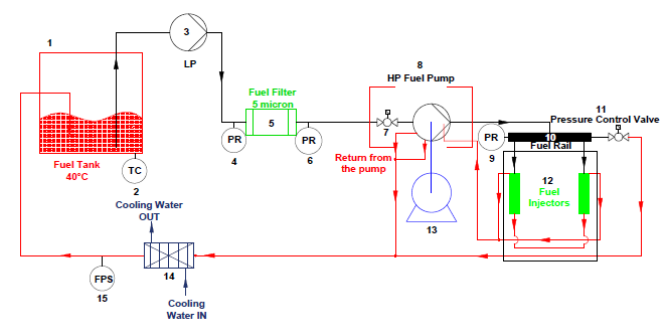


Figure 1. Simplified schematic representation of diesel fuel degradation rig.¹⁷

Table 1. Key for components labelled in Figure 1 above. All temperature measurements use K-Type thermocouples unless otherwise stated.

Label	Description	Label	Description
1	Fuel tank	9	Rail pressure sensor
2	Fuel tank temperature	10	Fuel Rail

3	Injection fuel lift pump	11	Rail pressure control valve (rail assembly)
4	Pre-Filter fuel pressure	12	Fuel injectors
5	Fuel filter	13	FIE pump drive motor (integrated in rig)
6	Post-Filter fuel pressure	14	Heat exchanger
7	Flow control valve (FIE pump assembly)	15	Fuel Property Sensor
8	HP fuel Common Rail pump		

A commercially available fuel injection system was modified comprising two solenoid diesel injectors, a common rail (CR), high pressure pump (HP), a lift pump (LP), a fuel filter, a fuel cooler and a fuel tank. A layout of the recirculation test rig designed for this study is shown in Figure 1 (for key refer Table 1). The rig also consisted of an on-board fuel property sensor (FPS), based on a tuning fork technology, which was used to directly measure physical properties such as dynamic viscosity, density, dielectric constant and temperature.

The FIE pump was driven by an electric motor with an in-line torque flange recording the motor speed and torque. Spill returns from the common rail, high pressure pump and injectors returned to the tank via a heat exchanger to maintain the fuel at 40 °C in the tank. To accelerate the aging process, injected fuel could also be recirculated and drawn before the lift pump.

The injectors were mounted in a custom-made aluminium block with integrated heater elements (cartridge heaters with internal temperature measurement) allowing the mounting temperatures to be set and controlled to simulate mounting within an actual engine cylinder head. The common rail pressure and injector actuation was controlled by a Stardex® common rail testing system which allowed selection of rail pressure up to 2000 bar and user-defined injection profiles (duration and frequency). Fuel temperature was measured at a number of points around the system in order to monitor the thermal cycle the fuel experienced. A control PC was used to operate the rig through which alarms and shutdowns could be defined.

Fuel Selection and Preparation

Two types of baseline diesel (B0) not treated with manufacturer-specific additive packs were used; one labelled as “EN590” which contained only refinery additives and the other, a commercial reference diesel labelled “RF79”. In Western Europe, FAME is commonly produced from rapeseed oil (RME) and soybean oil (SME). SME is known to have lower oxidation stability than RME due to its higher proportion of polyunsaturated compounds and was therefore selected for its increased propensity to oxidation.[25] The baseline diesels were then blended with FAME such that EN590 (B0) was mixed with a 10% v/v mixtures with SME while RF79 (B0) was blended with a 7% v/v mixture of SME and RME (SME:RME, 1:1 v/v). In order to ensure proper functioning of the diesel FIE, fuel properties must comply with the European specifications EN590 and can be blended with an EN14214 compliant FAME up to a maximum volume of 7%, but it is expected that this will increase to 10% in the coming years.[23] Therefore a 7-10% SME blend represents a realistic future diesel/FAME mixture.

In order to simplify the complexity involved in the diesel mixture to get a deeper understanding of the effects of the various components on fuel degradation and minimise the test time, a surrogate fuel, incorporating the general chemistries found in diesel, was used. As the majority of diesel sold in the EU contains FAME, this was incorporated. This surrogate fuel was made from mixing individual compounds (aromatics, a FAME compound (10% v/v), a cyclic hydrocarbon and an aliphatic hydrocarbon. A simple nitrogen heterocyclic compound (pyrrole) was included in a small quantity to represent a highly oxidative species.

Test Conditions

Prior to the commencement of each test, a flush cycles were carried out repeatedly with the test fuel for a defined period of time at reduced CR pressure and motor speed. In the FIE rig used in this study, fuel was continuously recirculated and underwent a periodic thermal cycle which accelerated the degradation process. As a representative temperature in a standard vehicle, the fuel temperature inside the tank was regulated to 40 °C. The tank was filled to about half of the capacity to allow air breathing while minimising system volume. The heating temperature of the two injector bodies was chosen to reflect in-use cylinder head temperatures while remaining safely below the auto-ignition temperatures of the test fuels used (256°C). The mounting temperature was consistent for all tests. The temperature was selected as a worst-case temperature of the cylinder head taking into account additional heat transfer generated by the combustion events. Each test was run continuously until a defined increase in the pressure drop (ΔP) across the filter was observed as ΔP was used as a measure of rate of solid deposition on the filter.

The matrix of experiments of this study is presented in Table 2. Tests 1 and 2 represented normal diesel fuel (EN590) with and without FAME (10% v/v SME) with active injection and injector heating and CR pressure at 2000 bar respectively. The effect of RF79, another type of normal diesel, was tested in the presence and absence of FAME (7% v/v SME+RME) in tests 3 and 4. Test 5 was carried out using a full component blended diesel fuel surrogate (FCB) which was prepared to represent EN590 with 10% FAME keeping similar reaction conditions. Test 6 was carried out as a baseline test with a minimal component blended surrogate fuel (MCB) comprising an aliphatic hydrocarbon and decalin cyclic hydrocarbon (D) in a 1:1 v/v mixture. In Test 7, the effect of FAME on fuel oxidation was tested by adding 10% v/v to MCB and tested under same experimental conditions. The effect of aromatics was investigated by mixing MCB with aromatic HC (1:1 v/v) (Ar) and trace (150 mgkg⁻¹) nitrogen containing heteroaromatic, pyrrole (Py), in test 8.

Table 2. Matrix of test fuels.

Test #	Fuel type	FAME content % vol.
1	EN590	-
2	EN590 + 10% FAME	10(SME)
3	RF79	-
4	RF79 + 7% FAME	7(RME+SME)
5	Full Component Blend (FCB) – MCB+Ar+MO+Py	10
6	Minimum Component Blend (MCB) – aliphatic HC+D	-
7	MCB + 10% FAME	10
8	MCB + Ar + Py	-

Results and Discussion

Effect of FAME on fuel degradation

It is well documented that the inclusion of FAME within EN590 diesel has led to a reduction in the stability of the blend. Studies have shown that increasing the degree of unsaturation leaves FAME susceptible to oxidation via a radical mechanism.[4, 7] Recent studies assessing the oxidation stability of diesel and biodiesel blends using a PetroOxy device (as per ASTM D7545 and EN16091) have concluded that the addition of 10% FAME (RME) reduced the induction period of the blend by 2.6 times (at 403K) compared with the baseline B0 diesel fuel indicating that FAME had a significant detrimental effect on the oxidation stability of the fuel.[21] The PetroOxy test exposes the fuel to elevated temperatures under a pressurised oxygen environment. Despite the findings related to the variations in stability of B0 and B10 blends, it is not clear if these differences would also be observed under realistic operating conditions within a vehicle fuel injection system.

Standard mineral diesel testing

The impact of FAME on fuel degradation and filter loading, at 2000 bar with heated active injectors, was investigated as solid deposition in the filter has a direct impact on engine operability. Figures 2 and 3 show the increase in filter differential pressure with time for two types of standard diesel fuels, EN590 (Figure 2) and RF79 (Figure 3) with and without FAME. The addition of FAME to the diesel fuel did not result in the significant reduction in stability (characterised by a reduced induction period or rate of deposition).[21]

While there is a slight variation in the rate of increase of ΔP between the two fuels, observed from 6000 to 8000 minutes (Figure 2), the magnitude of the difference is small and within the bounds of experimental accuracy. Induction periods for both fuels lasted for approximately 2100 minutes with a very similar rate of subsequent deposition.

The RF79 tests were run for reduced durations to prevent component exposure to highly degraded fuel and prolong hardware life. Based on the results shown in Figure 3, it was determined that a ΔP change of 0.06 bar would be sufficient to indicate fuel degradation and, therefore, tests were ended when this ΔP was achieved. RF79 results show an increase both in the presence and absence of FAME with the induction period for both fuels lasting approximately 1800 minutes with a similar rate of solid deposition.

The pressure drop was measured across the filter, to demonstrate the level of filter blocking in the system, and hence the particle deposition. The resolution of the pressure transducers in the pre and post fuel filter locations was 0.01 bar. An “averaged data plot” approach was adopted wherein the data points at each ΔP value was averaged (by mean) which is denoted by the dot in the middle. The horizontal error bars denote the range in time for a particular ΔP . The horizontal range bars from these runs were calculated as the shortest time taken to achieve specific ΔP values.

The fact that diesel degrades in the absence of FAME, would suggest that other, non-FAME, diesel components contribute to variations in the oxidative stability of the bulk fuel under accelerated test conditions which, in turn, leads to solid formation and filter deposits. It is not possible to ascertain which diesel component has the greatest impact on stability, therefore additional investigations are required to

examine these in isolation.

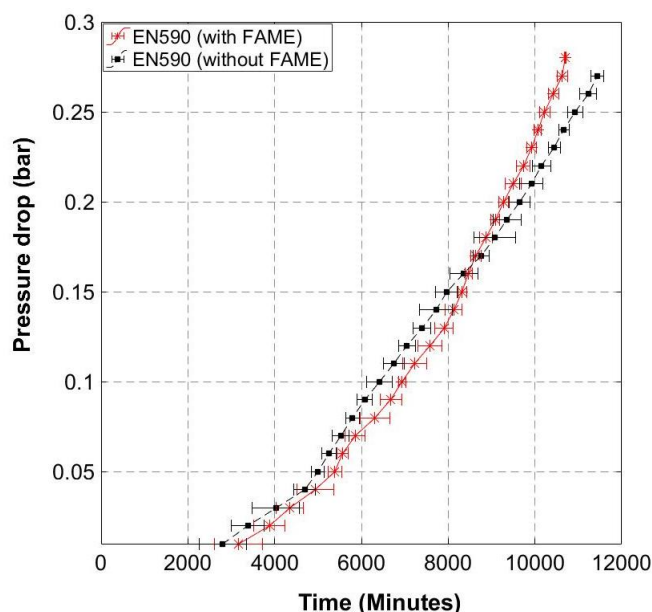


Figure 2. Comparison of fuel degradation in the presence and absence of FAME of petroleum diesel EN590. Reaction conditions: EN590 (B0) and with 10% FAME (B10), CRP: 2000 bar, Injection event: On, Heaters: On (200°C).

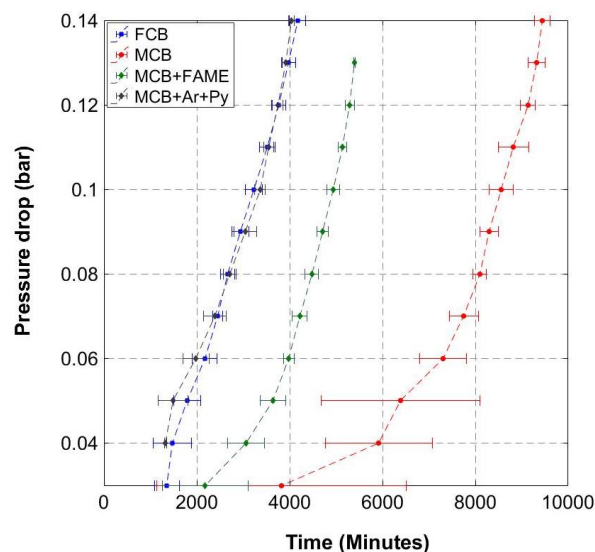


Figure 4. Pressure drop (ΔP) across the fuel filter comparison of the surrogate FCB, baseline test (MCB), MCB+FAME, MCB+Ar+Py (Table 2). Reaction conditions: CRP: 2000 bar, Injection event: On, Heaters: On (200°C).

FCB represents a reasonable facsimile to diesel (Table 2). The surrogate fuel undergoes degradation rapidly with a lag time of 1,800 minutes before the ΔP is seen to increase, finally resulting in a ΔP of 0.14 bar in approximately 4,100 minutes of continuous running. The effect of aliphatic HCs in the absence of other chemically active species was tested using a baseline mixture (MCB). Interestingly the fuel did degrade and produced filter deposits, albeit far slower than FCB, with a lag time of approximately 2.3x longer and taking approximately 9,800 minutes to reach a $\Delta P=0.14$ bar. While far less prone to oxidation than the other species, cycloalkanes are known to undergo oxidation, though complicated, via hydrogen abstraction at such high pressures and temperatures.[26]

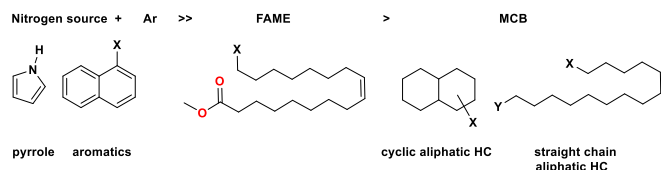
To investigate the impact of FAME on the deposit formation, 10% v/v FAME was blended with MCB. The presence of FAME accelerated the oxidation compared to the baseline test (MCB) but demonstrated a longer lag time and slower rate than the FCB test. Due to the lower lubricity of the blend (as a result of the lack of aromatics), the reaction could not be run to completion and was terminated earlier. However, the presence of FAME alone in the fuel did not result in the same rate of solid deposition on the filter as FCB.

Both MCB and MCB+FAME tests demonstrated slower solid deposition compared to FCB test suggesting that the aromatics (Ar) and hetero-aromatics (Py) played a vital role in accelerating the deposition process. To examine the effect of aromatic species on fuel degradation, 150 mgkg⁻¹ of pyrrole (Py) was added to the fuel mixture (MCB: aromatic HC, 1:1 v/v). The MCB+Ar+Py test was found to be similar to FCB. The lag time and rate of deposition is within error of FCB, suggesting that aromatic species in diesel fuel acts as the initiator in fuel oxidation resulting in solid formation.

Diesel surrogate fuel testing

It was important to investigate the role of all components present in the fuel, including those in trace quantities, on fuel oxidation and filter blocking to get a deeper understanding on the mechanisms involved in fuel degradation. Therefore, further testing was performed using synthetic surrogates of diesel. A series of four different blends were assessed with different components added to base fuel (MCB). These were a “Full component blend” (FCB) test,

Each of the components in fuel, aliphatic HCs, Ar, FAME and Py, are unstable and are known to oxidise with one of the components in the fuel acting as an instigator. Although the lag times differed, the deposition rate appeared to be approximately the same across all species. This suggests the following susceptibility to oxidation:



This demonstrates that while FAME is commonly thought to be highly susceptible to oxidation, other species present in the fuel can also undergo chemical transformations at such high pressures and temperatures and thus contribute to deposit formation.

Conclusions

A bespoke fuel degradation rig, mimicking a light duty diesel, was designed and commissioned and used to investigate the impact of accelerated test conditions and fuel composition on deposit formation and filter loading. Contrary to the findings of standard oxidation stability tests, in particular the PetroOxy test, the addition of 10% v/v FAME was found to have no impact on the rate of filter deposition and associated pressure differential using both EN590 and RF79 standard diesel fuels. Using a surrogate B10 diesel fuel mixture, which was used to replace standard petroleum diesel, demonstrated that biodiesel was not the most significant cause of degradation. The surrogate fuel findings suggest that the least stable component acts as an instigator for the degradation process with FAME being more stable than other trace constituents.

The studies, undertaken at accelerated conditions to represent more realistic environmental conditions, demonstrate that FAME is not solely responsible for fuel degradation. Other components, such as aromatics or nitrogenous species, present in the fuel have a greater impact on diesel fuel stability.

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Contact Information

Dr. Kesavan Gopalan CChem MRSC
Email: K.Gopalan@bath.ac.uk

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Definitions/Abbreviations

ΔP	delta P (pressure drop)
Ar	Aromatic HC
Bx	Biodiesel blend ratio (x=0, (i.e. B0) means no biodiesel, x=10 (i.e. 10) means 10 per cent biodiesel in diesel etc.)

CR
CRP
D
FCB
FIE
FAME
HC
HEFCE

HEIF

HP
HPCR
IP
LP
MCB
OEM

Py
RME
SME
UoB

Common Rail
Common Rail Pressure
Decalin
Full Component Blend
Fuel Injection Equipment
Fatty Acid Methyl Ester
Hydrocarbons
Higher Education Funding Council of England
Higher Education Innovation Fund
High Pressure pump
High Pressure Common Rail
Induction Period
Lift Pump
Minimal Component Blend
Original Equipment Manufacturer
Pyrrole
Rapeseed Methyl Ester
Soyabean Methyl Ester
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